

PATENT ABSTRACTS OF JAPAN

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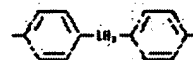
(54) MODIFIED POLYESTER FIBER MATERIAL AND ITS PRODUCTION

(57)Abstract:

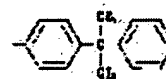
PURPOSE: To obtain a modified polyester fiber material excellent in durable antimicrobial or/and moisture absorption and desorption, water absorption, etc., by carrying out the graft copolymerization of the surfaces of polyester fibers with collagen or/and an antimicrobial agent.



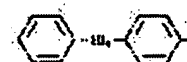
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**FY**

CONSTITUTION: The method for readily producing a modified polyester fiber material is obtained by applying a treating liquid prepared by adding a compound having radical-polymerizable double bond, e.g. a monomer, expressed by formula I {R is a directly bound formula II to IV or C_nH_{2n} [(n) is 1-6]; Z is H or CH_3 ; (a) and (b) are [(a)+(b)]=0-30; (x) and (y) are each 0 or a positive integer so as to provide [(x)+(y)]=0-30; [(a)+(b)+(x)+(y)] \geq 10}, containing a polyoxyalkylene group having ≥ 1000 molecular weight or further a monomer containing OH, COOH, NH_2 , sulfo group, phosphate group, etc., and a monomer, etc., containing aziridine group to an aqueous solution of atherocollagen as a collagen or/and an antimicrobial agent to the surface of polyester fibers, heat-treating the resultant fibers and carrying out the graft copolymerization. The resultant modified polyester material has antimicrobial, antistatic and stain-proofing properties rich in durability.

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CLAIMS

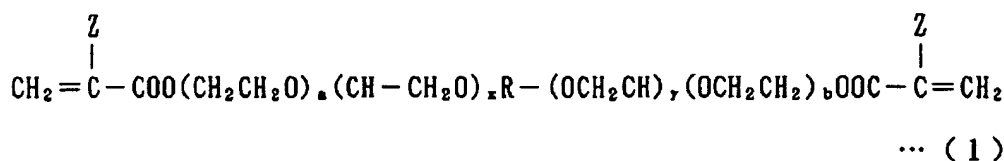
[Claim(s)]

[Claim 1] The refining polyester fiber ingredient characterized by the collagen or/and the antimicrobial agent being combined by graft polymerization on the surface of polyester fiber.

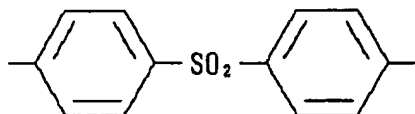
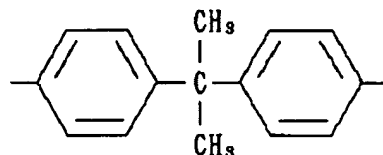
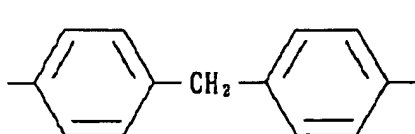
[Claim 2] Textile materials according to claim 1 whose antimicrobial agent is the compound which has the double bond in which a radical polymerization is possible.

[Claim 3] The manufacture approach of the refining polyester fiber ingredient characterized by carrying out the polymerization of it after giving the processing liquid obtained by adding the monomer to which molecular weight includes two or more double bonds in which a radical polymerization is possible including 1000 or more polyoxyalkylene groups in a collagen or/and an antimicrobial agent to a polyester fiber ingredient.

[Claim 4] 2 functionality monomer shown in a collagen or/and an antimicrobial agent by the following component ** - **.* following general formula (1), [Formula 1]



[上式中、Rは直接結合された



または $-\text{C}_n\text{H}_{2n}-$ （ここで n は1～6の整数を表す）を表し、ZはHまたは $-\text{CH}_3$ を表し、 a および b は $a+b$ が0～50となるような正の整数を表し、 x および y は $x+y$ が0～30となるような0または正の整数を表す。
但し、 $a+b+x+y$ は10以上であるものとする。]

** The manufacture approach of the refining polyester fiber ingredient characterized by carrying out the polymerization of it after giving the processing liquid obtained by adding the multifunctional compound containing the monomer containing a hydroxyl group, a carboxyl group, an amino-group sulfonic group, or a phosphoric-acid radical and the monomer containing one ** aziridine radical, or two or more aziridine radicals to a polyester fiber ingredient.

[Claim 5] The approach according to claim 3 or 4 an antimicrobial agent is the compound which has the double bond in which a radical polymerization is possible.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the refining polyester fiber ingredient which has antibacterial or/and moistness and also has absorptivity, electrostatic nature, and antifouling property.

[0002]

[Description of the Prior Art] Various bacteria and mold exist in our perimeter. an elevated temperature -- under a humid environment, especially, it becomes active, and it is invaded by athlete's foot, the phenomenon of putrefaction or fermentation is caused, or those propagation generates an unpleasant odor. sweat -- it depends on bacteria's operation of that a stinking smell also arises. Bacteria breed in the sweat absorbed by the sweat on the front face of the skin, underwear, socks, etc., and it becomes a stinking cause of a thing that there is no odor in sweat itself.

[0003] The evil by the microorganism has adverse effects, such as embrittlement, discoloration, an infective disease, a dermatosis, and athlete's foot, not only to an offensive odor but to the body and clothing. In order to prevent the evil by these microorganisms, the antibacterial treatment of textile materials is considered variously. For example, with various kinds of composition or semi-synthetic fiber yarn, to the approach of giving an antimicrobial agent by scour lump in the phase of spinning, and the natural fiber, the approach like a publication and the approach of forming a copper chelate for acrylonitrile system fiber are further learned by JP,57-51874,A, and it is provided with what can be satisfied to some extent.

[0004] However, since it does not have a reaction radical, the aforementioned approach cannot be used but the method (JP,3-76871,A) of making the chitin system matter contain in the state of distribution in resin is learned for polyester fiber. However, by the approach of JP,3-76871,A, since the chitin system matter was only distributing in resin, the chitin system matter was eluted, or the resin containing the chitin system matter itself was omitted from on fiber, and the product obtained was inferior to endurance with wash.

[0005] Moreover, JP,61-22070,B and JP,62-60509,B are made to carry out the chemical bond of the polyester fiber and the antimicrobial agent which gave the reaction radical, and it is proposed that this gives an antimicrobial agent. However, it has the problem that it becomes [a processing process] long and is complicated since it is a two-step art, although this approach can give a durable product, and processing cost also goes up, and the endurance of the product obtained cannot say that it is enough to industrial wash, either.

[0006] Furthermore, the approach of giving absorptivity, an antielectricity characteristic, and antifouling property is conventionally learned as refining processing to polyester fiber. These processings are for improving the property of the polyester fiber made into demerit compared with the natural fiber.

However, in the state of [humid] **** which a natural fiber has, moisture was not able to be inhaled by this processing, and the polyester fiber which has the moisture absorption and desorption characteristics of emitting moisture in dryness was not able to be obtained.

[0007]

[Problem(s) to be Solved by the Invention] Therefore, this invention solves the trouble of the conventional technique like the above, and makes it a technical problem to offer the polyester fiber ingredient which has antibacterial [durable] or/and moisture absorption and desorption characteristics, absorptivity, an antielectricity characteristic, and antifouling property, and the method of manufacturing such a polyester fiber ingredient easily and cheaply industrially in a list.

[0008]

[Means for Solving the Problem] In order to solve the above-mentioned technical problem, this invention offers the refining polyester fiber ingredient characterized by the collagen or/and the antimicrobial agent being combined by graft polymerization on the surface of polyester fiber. This invention offers the manufacture approach of the refining polyester fiber ingredient characterized by carrying out a polymerization, after giving the processing liquid obtained by adding the monomer to which molecular weight includes two or more double bonds in which a radical polymerization is possible including 1000 or more polyoxyalkylene groups in a collagen or/and an antimicrobial agent again to a polyester fiber ingredient.

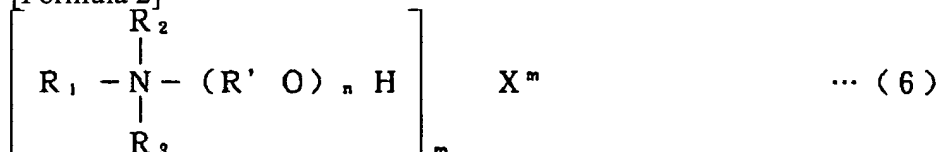
[0009] The polyester fiber ingredient used for this invention may be in what kind of gestalt of rose hair, a tow, yarn, etc. textiles, knitting, a nonwoven fabric, etc. Moreover, you may be what kind of things, such as plain weave, twill, satin, ***** and ***** , also about a textile construction or knitted tissues, such as textiles and knitting. Moreover, you may be what kind of things, such as non-twisted yarn, strong throwing, and structure finished yarn, also about the yarn usage, and may be interweaving with other natural fibers and a chemical fiber, mix spinning, and union.

[0010] As a collagen, TOPOKORAGEN by which pulverization was carried out, its distributed solution, the water solution of atelocollagen, or the colloidal solution can be used, and the water solution of atelocollagen is preferably used from a viewpoint of the endurance of the moistness acquired. The collagen existed during the organization of the skin of an animal, a bone, a tendon, a gear tooth, a blood vessel, intestines, an eye, etc., and it is the protein which is playing protection of a living organism, and the role of gestalt maintenance of each organization, and the collagen molecule (tropocollagen) consisted of three polypeptide chains, and has taken spiral structure. If an enzyme etc. removes the telopeptide which exists in the end of this tropocollagen molecule, it will come to dissolve in a protein modifier water solution, aqueous acids, etc. The collagen which removed the telopeptide is called atelocollagen and is a with die length of 2800A, and a molecular weight of about 300,000 thing.

[0011] Moreover, as an antimicrobial agent, a quarternary-ammonium-salt mold surfactant, GUANAIDO system drugs, an animal system high molecular compound, etc. can be mentioned. A following-type (6) compound can be mentioned as an example of a quarternary-ammonium-salt mold surfactant.

[0012]

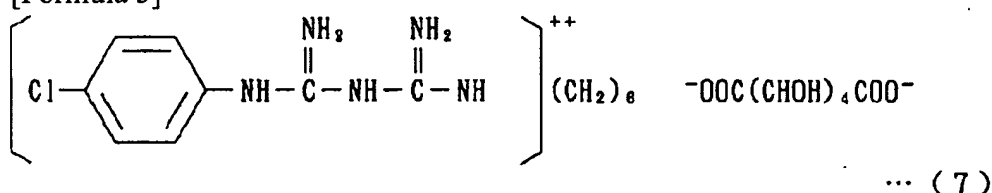
[Formula 2]



[0013] A following-type (7) compound can be mentioned as an example of GUANAIDO system drugs.

[0014]

[Formula 3]

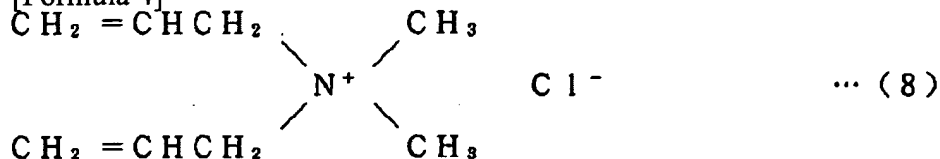


[0015] Chitosan can be mentioned as an animal system high molecular compound. Chitosan deacetylates the chitin which is the naturally-occurring polymers which exist in frames, shells, etc. of Mollusca, such as crustaceans, such as a shrimp, and ****, a krill, at **. Moreover, although the gestalt of chitosan may be in which condition of impalpable powder, dispersion liquid, and a water solution, especially, it quarternary-ammonium-salt-izes and what was used as the water solution is used more preferably than the viewpoint of antibacterial and its endurance.

[0016] Furthermore, if the antimicrobial agent which has double association in which the radical polymerization of the various above-mentioned antimicrobial agents is possible is used, endurance will improve further. A following-type (8) compound can be mentioned as an example of such an antimicrobial agent.

[0017]

[Formula 4]

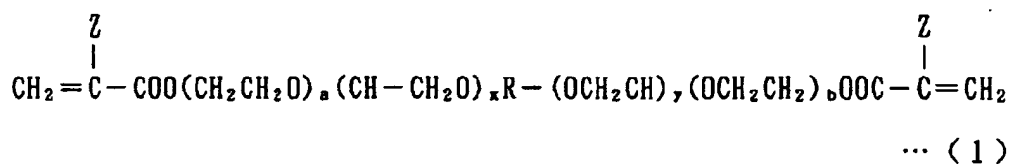


[0018] Next, the approach of this invention for manufacturing the above-mentioned textile materials is explained to a detail. The water solution of a monomer with which molecular weight includes two or more double bonds in which a radical polymerization is possible including 1000 or more polyoxyalkylene groups in an above-mentioned collagen or/and an above-mentioned antimicrobial agent is prepared.

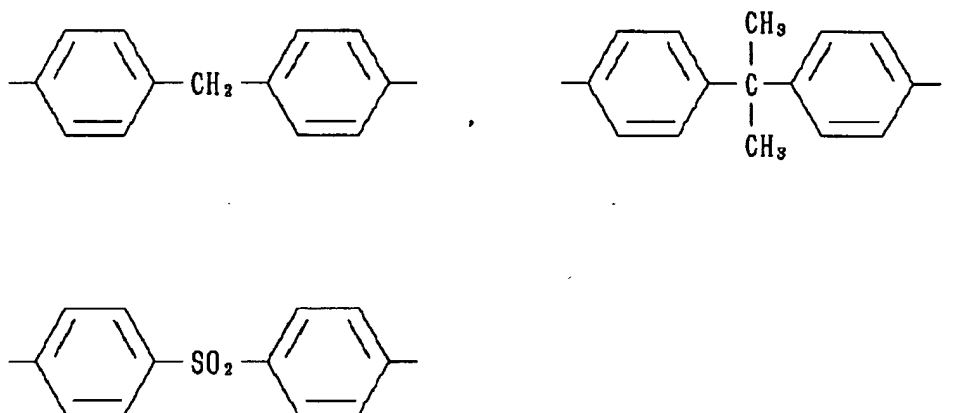
[0019] Molecular weight can mention compound ** of the following general formula (1) including 1000 or more polyoxyalkylene groups, for example as a monomer which has two or more double bonds in which a radical polymerization is possible.

[0020]

[Formula 5]



[上式中、Rは直接結合された

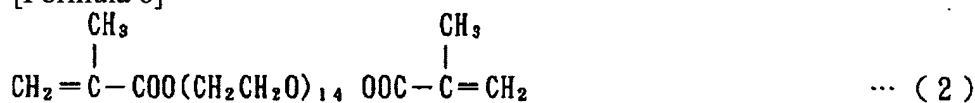


または $-\text{C}_n\text{H}_{2n}-$ （ここで n は1～6の整数を表す）を表し、 Z は H または $-\text{CH}_3$ を表し、 a および b は $a+b$ が0～50となるような正の整数を表し、 x および y は $x+y$ が0～30となるような0または正の整数を表す。但し、 $a+b+x+y$ は10以上であるものとする。]

[0021] Moreover, the compound of the following type (2), (3), (4), and (5) can be mentioned as the example.

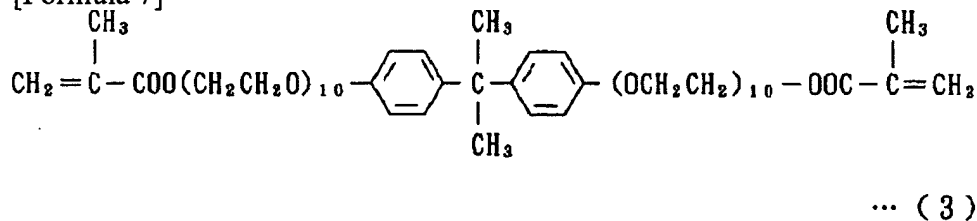
[0022]

[Formula 6]



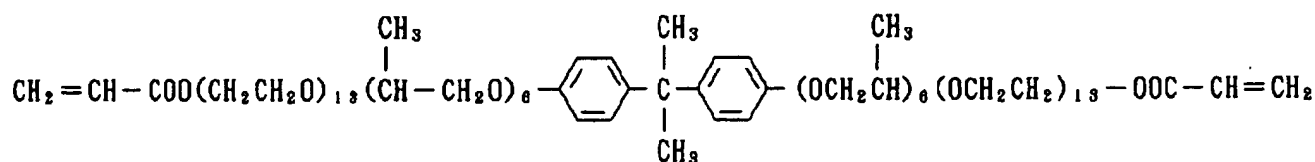
[0023]

[Formula 7]



[0024]

[Formula 8]



... (4)

[0025]

[Formula 9]



... (5)

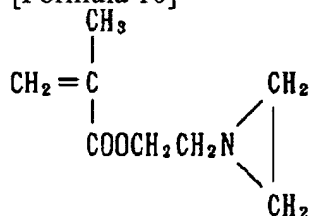
[0026] Furthermore, in order to make it react smoothly or to make it join together more firmly, the multifunctional compound containing the monomer or two or more aziridine radicals which contain the monomer and one ** aziridine radical containing radical polymerization catalyst, ** hydroxyl group and a carboxyl group, the amino group, a sulfonic group, or a phosphoric-acid radical in said water solution may be added. The mixed ratio of component **, **, **, and an antimicrobial agent + collagen is weight, and it is good that it is good that it is the range of 1:0.1-1:0.01-1:0.01-1, and it is [as opposed to / as opposed to / with the padding method / 1 - 20 % of the weight / at dip coating / in the amount of component ** in a water solution / fiber weight] 1 - 20% of the weight of an amount.

[0027] A peroxide and an azo compound can be used as radical polymerization catalyst. As a monomer containing a hydroxyl group, a carboxyl group, or the amino group, an acrylic acid and a methacrylic acid can be mentioned, for example. Moreover, it is also possible to use the monomer which has a sulfonic group or a phosphoric-acid radical instead of the monomer containing a hydroxyl group, a carboxyl group, or the amino group. However, since it may combine with the antimicrobial agent which is cationicity in this case and a gum rise may be carried out in water-solution liquid, caution is required. However, since it also has the operation whose collagen controls a gum rise when using a collagen together, the activity is possible enough.

[0028] As a multifunctional compound containing the monomer or two or more aziridine radicals containing one aziridine radical, the compound of the following formula (9), (10), and (11) can be mentioned, for example.

[0029]

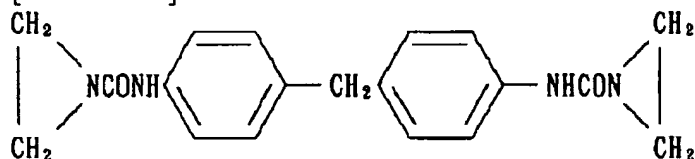
[Formula 10]



... (9)

[0030]

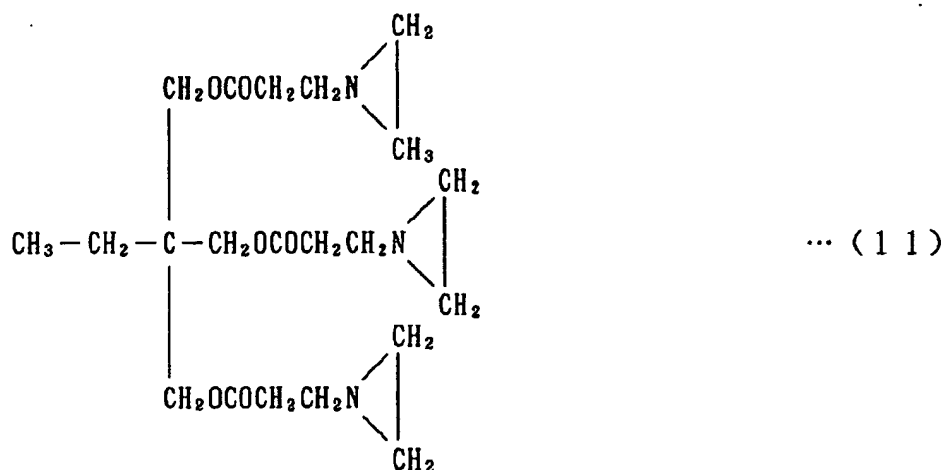
[Formula 11]



... (1 0)

[0031]

[Formula 12]



[0032] Furthermore, when the above-mentioned monomer mixture is not soluble at water, a surfactant and an organic solvent may be added. Next, the above-mentioned water solution is given to textile materials by the padding method etc. After giving a water solution to textile materials, dry heat treatment, steaming processing, ultraviolet rays and microwave, or electron beam irradiation is made to perform graft polymerization. Moreover, although some utilization ratios of a monomer fall, they can also carry out graft polymerization by the uptake method.

[0033] After a polymerization reaction is completed, rinsing, hot water rinsing, etc. are performed, the unreacted matter is removed, it dries and the finishing set for specification doubling and wrinkling picking is performed if needed.

[0034]

[Effect of the Invention] The refining polyester fiber ingredient of this invention has durable antibacterial ability or/and moisture absorption and desorption characteristics by carrying out graft polymerization to the antimicrobial agent or/and the collagen. Therefore, when it is used for underwear, a blouse, etc., it does not have adverse effects, such as embrittlement of a textile, discoloration, an infective disease, a dermatosis, and athlete's foot, to the offensive odor by the microorganism, and clothing by antibacterial ability.

[0035] Moreover, with the moisture absorption and desorption characteristics of a collagen, displeasure, such as a feeling of MURE, is abolished and the comfortable raw material which also has an antielectricity characteristic, absorptivity, and antifouling property further can be offered. Furthermore, since it had joined together more firmly, endurance improves and the textile materials with which antibacterial ability does not fall after industrial wash could be offered. Therefore, it can be used for the various clothes for the hospital infection prevention by MRSA (MESHICHIRIN-proof Staphylococcus aureus) which poses a problem recently, a sheet, a curtain, etc.

[0036]

[Example] Hereafter, an example explains this invention further. In addition, each performance evaluation in an example was performed by the following approach.

(1) Antibacterial number-of-microorganism measuring method (textiles sanitary finishing conference)

[0037] (2) Weight $A=(b-a)/aB=(c-a)/a$ after leaving it for 30 minutes in the chamber of the weight of $c=35$ degrees C after leaving it for 30 minutes in the chamber of the condition of moisture-absorption-and-desorption-characteristics the weight of $b=20$ degrees C after drying in the dryer of $a=110$ degrees C of count by the degree for 1 hour and 65% of humidity RH, and the condition of 95% of humidity RH

[0038] (3) Antielectricity characteristic JIS L-1094 It is (4) absorptivity dropping test B law. JIS L-1096 It is (5) antifouling-property diamond mull technique (Japan Chemical Fibres Association specification JCFA TM-104) A law.

[0039] (6) Wash approach JIS L-0217 Using ZABU (Kao make) 5 g/l, meta-sodium silicate 5 g/l, and fault sodium carbonate 2 g/l as a (7) industrial wash approach detergent, wash was performed for 40 minutes at 60 degrees C, and then it rinsed for 15 minutes with chilled water, then, dried at 80 degrees C

with the tumbler type dryer, and took 103 law for one count of wash.

[0040] After carrying out impregnation of the following water solution to example 1 polyester 100% crepe de Chine (75d - 72f) and extracting to 65% of rates of impregnation with a mangle, steam heat treatment was performed for 10 minutes at 110 degrees C, and molten-bath washing, desiccation, and a finishing set were performed. The engine performance of the obtained textile is described in a table 1. Compound of a formula (2) 4.0 % of the weight Methacrylic acid 0.5 % of the weight Compound of a formula (9) 0.5 % of the weight Atelocollagen water solution (concentration 10%) 3.0 % of the weight Chitosan water solution 3.0 % of the weight (concentration 10%, quarternary ammonium salt) Ammonium persulfate 0.5 % of the weight [0041] After carrying out impregnation of the following water solution to example of comparison 1 polyester 100% crepe de Chine (75d - 72f) and extracting to 65% of rates of impregnation with a mangle, dry heat treatment was performed for 1 minute at 150 degrees C, and molten-bath washing, desiccation, and a finishing set were performed. The engine performance of the obtained textile is described in a table 1.

ERASU TRON W-11 4.0 % of the weight (water-soluble polyurethane resin, product made from the first industry)

ERASU TRON cat 64 0.5 % of the weight (a catalyst, product made from the first industry)

Chitosan water solution 3.0 % of the weight (concentration 10%, quarternary ammonium salt)

Sodium bicarbonate 0.01 % of the weight [0042] After carrying out impregnation of the following water solution to the example 2 polyester 100% twill (structure finished yarn, 75d - 36f) and extracting to 70% of rates of impregnation with a mangle, it rolled round on the roll using the microwave processor, performing a microwave exposure by output 4kw, and the exposure was performed for 5 more minutes. Next, molten-bath washing, desiccation, and a finishing set were performed. The engine performance of the obtained textile is described in a table 1.

[0043]

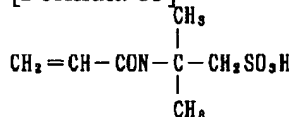
Compound of a formula (3) 5.0 % of the weight Methacrylic acid 0.5 % of the weight Compound of a formula (10) 0.5 % of the weight Atelocollagen water solution (concentration 10%) 3.0 % of the weight Compound of a formula (6) 1.0 % of the weight V-50 (the product made from the Wako Pure Chem industry, azo system catalyst) 0.5 % of the weight [0044]

After carrying out impregnation of the following A water solution to the example of comparison 2 polyester 100% twill (structure finished yarn, 75d - 36f) and extracting to 50% of rates of impregnation with a mangle, it rolled round on the roll using the microwave processor, performing a microwave exposure by output 4kw, and the exposure was performed for 5 more minutes. Next, after carrying out impregnation of the following B water solution and extracting it to 80% of rates of impregnation with a mangle, heat treatment was performed for 1 minute at 150 degrees C, and molten-bath washing, desiccation, and a finishing set were performed. The engine performance of the obtained textile is described in a table 1.

[0045]

A water solution Compound of a formula (3) 5.0 % of the weight Methacrylic acid 0.5 % of the weight Following-type compound 1.0 % of the weight V-50 (the product made from the Wako Pure Chem industry, azo system catalyst) 0.5 % of the weight [0046]

[Formula 13]



[0047]

B water solution Compound of a formula (6) After carrying out impregnation of the following water solution to 2.0 % of the weight example 3 polyester 100% taffeta (75d - 72f) and extracting to 30% of rates of impregnation with a mangle, the electron ray of 2Mrad was irradiated using the electron ray processor. Next, molten-bath washing, desiccation, and a finishing set were performed. The engine performance of the obtained textile is described in a table 1.

[0048]

The compound of a formula (4) 4.5 % of the weight Methacrylic acid 2.0 % of the weight The compound of a formula (11) 1.0 % of the weight Atelocollagen water solution (concentration 10%) 3.0 % of the weight The compound of a formula (7) 3.0 % of the weight Potassium persulfate 0.5 % of the weight [0049] Liquid flow dyeing equipment was used for example 4 polyester 100% knitting (****, 75d - 36f), and processing was carried out for 30 minutes at 100 degrees C in the following water solution. Then, desiccation and a finishing set were performed. The engine performance of the obtained textile is described in a table 1.

The compound of a formula (2) 6.0 % of the weight Methacrylic acid 2.0 % of the weight The compound of a formula (9) 2.0 % of the weight Atelocollagen water solution (concentration 10%) 3.0 % of the weight The compound of a formula (8) 1.0 % of the weight Ammonium persulfate 0.5 % of the weight [0050] After carrying out impregnation of the following water solution to example 5 polyester 100% crepe de Chine (75d - 72f) and extracting to 65% of rates of impregnation with a mangle, steam heat treatment was performed for 10 minutes at 110 degrees C, and molten-bath washing, desiccation, and a finishing set were performed. The engine performance of the obtained textile is described in a table 1.

Compound of a formula (2) 4.0 % of the weight 2-hydroxyethyl methacrylate 0.5 % of the weight Compound of a formula (9) 0.5 % of the weight Collagen impalpable powder (particle diameter 2micro) 3.0 % of the weight Compound of a formula (8) 1.0 % of the weight V-50 (the product made from the Wako Pure Chem industry, azo system catalyst) 0.5 % of the weight [0051] After carrying out impregnation of the following water solution to example 6 polyester 100% crepe de Chine (75d - 72f) and extracting to 65% of rates of impregnation with a mangle, steam heat treatment was performed for 10 minutes at 110 degrees C, and molten-bath washing, desiccation, and a finishing set were performed. The engine performance of the obtained textile is described in a table 1.

Compound of a formula (2) 4.0 % of the weight Dimethylaminoethyl methacrylate 0.5 % of the weight Compound of a formula (9) 0.5 % of the weight Compound of a formula (6) 1.0 % of the weight V-50 (the product made from the Wako Pure Chem industry, azo system catalyst) 0.5 % of the weight [0052] [A table 1]

表 1

| | | 未加工 布 帛 | 実施 例 1 | 比較例 1 | 実施 例 2 | 比較 例 2 | 実施 例 3 | 実施 例 4 | 実施 例 5 | 実施 例 6 |
|-------------|------|------------|-----------|----------|-----------|-----------|-----------|-----------|-----------|-----------|
| 抗 菌 性 | 初期 | 0.2 | 4.4 | 4.4 | 4.4 | 4.4 | 3.8 | 4.4 | 4.2 | 4.2 |
| | 洗濯後 | — | 3.8 | 1.2 | 4.4 | 2.3 | 3.0 | 3.8 | 4.0 | 3.6 |
| | 工業洗濯 | — | 1.8 | 0.3 | 3.9 | 0.9 | 2.5 | 3.5 | 3.7 | 2.8 |
| 吸 放 湿 | 初期 | A | 0.4 | 0.7 | — | 0.8 | — | 0.6 | 0.7 | 0.7 |
| | | B | 0.5 | 2.5 | — | 2.8 | — | 2.3 | 2.7 | 2.6 |
| 制 電 性 | 初期 | 6300 | 120 | 160 | 180 | 350 | 200 | 140 | 150 | 240 |
| | 洗濯後 | — | 400 | 2100 | 300 | 300 | 800 | 280 | 500 | 700 |
| | 工業洗濯 | — | 400 | 6800 | 400 | 300 | 900 | 300 | 500 | 900 |
| 吸 水 性 | 初期 | 60以上 | 4 | 5 | 瞬間 | 4 | 28 | 瞬間 | 4 | 18 |
| | 洗濯後 | — | 7 | 60以上 | 瞬間 | 瞬間 | 56 | 瞬間 | 8 | 26 |
| | 工業洗濯 | — | 8 | 60以上 | 瞬間 | 瞬間 | 58 | 瞬間 | 8 | 28 |
| 防 汚 性 | 初期 | 1 | 4 | 2 | 4 | 3 | 4 | 4 | 4 | 4 |
| | 洗濯後 | — | 4 | 1-2 | 3-4 | 3 | 4 | 4 | 4 | 4 |
| | 工業洗濯 | — | 4 | 1-2 | 3-4 | 4 | 4 | 4 | 4 | 4 |

洗濯後、工業洗濯は20回後

[Translation done.]